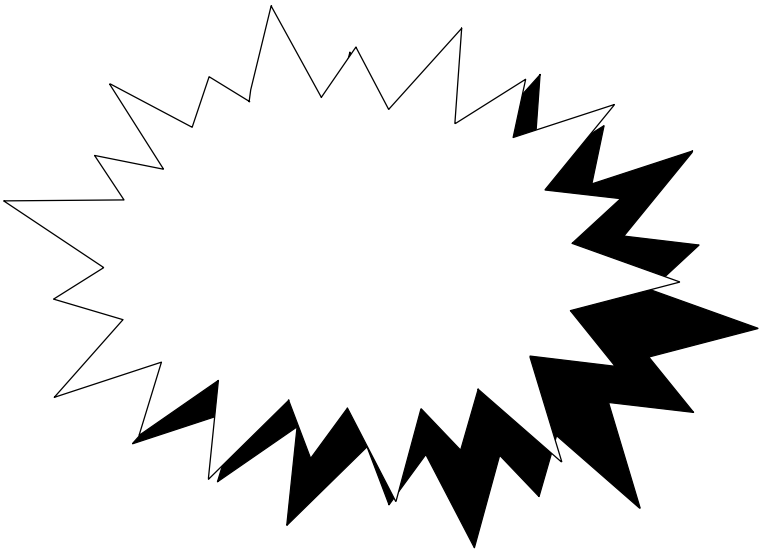

Chemical Analysis and Testing Task

Laboratory Analytical Procedure

LAP-005

Procedure Title: Standard Method for Ash in Biomass

Author: Tina Ehrman

Date: 4/28/94

ISSUE DATE: 4-28-94 **SUPERSEDES:** 8/18/92

DISCLAIMER

These Standard Biomass Analytical Methods (“Methods”) are provided by the National Renewable Energy Laboratory (“NREL”), which is operated by the Midwest Research Institute (“MRI”) for the Department Of Energy.

Access to and use of these Methods shall impose the following obligations on the user. The user is granted the right, without any fee or cost, to use, copy, modify, alter, enhance and distribute these Methods for any purpose whatsoever, except commercial sales, provided that this entire notice appears in all copies of the Methods. Further, the user agrees to credit NREL/MRI in any publications that result from the use of these Methods. The names NREL/MRI, however, may not be used in any advertising or publicity to endorse or promote any products or commercial entity unless specific written permission is obtained from NREL/MRI. The user also understands that NREL/MRI is not obligated to provide the user with any support, consulting, training or assistance of any kind with regard to the use of these Methods or to provide the user with any updates, revisions or new versions.

THESE METHODS ARE PROVIDED BY NREL/MRI "AS IS" AND ANY EXPRESS OR IMPLIED WARRANTIES, INCLUDING BUT NOT LIMITED TO, THE IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE ARE DISCLAIMED. IN NO EVENT SHALL NREL/MRI BE LIABLE FOR ANY SPECIAL, INDIRECT OR CONSEQUENTIAL DAMAGES OR ANY DAMAGES WHATSOEVER, INCLUDING BUT NOT LIMITED TO CLAIMS ASSOCIATED WITH THE LOSS OF DATA OR PROFITS, WHICH MAY RESULT FROM AN ACTION IN CONTRACT, NEGLIGENCE OR OTHER TORTIOUS CLAIM THAT ARISES OUT OF OR IN CONNECTION WITH THE ACCESS, USE OR PERFORMANCE OF THESE METHODS.

Standard Method for Ash in Biomass

Laboratory Analytical Procedure #005

1 Introduction

1.1 This procedure has been adopted by ASTM as an ASTM Standard Test Method for the determination of ash in biomass.

2. Scope

2.1 This test method covers the determination of ash, expressed as the percentage of residue remaining after dry oxidation (oxidation at 550 to 600^{o}C), of hard and soft woods, herbaceous materials (such as switchgrass and sericea), agricultural residues (such as corn stover, wheat straw, and bagasse), wastepaper (such as office waste, boxboard, and newsprint), acid and alkaline pretreated biomass, and the solid fraction of fermentation residues. All results are reported relative to the 105^{o}C oven-dried weight of the sample.

2.2 All analysis shall be performed according to the Ethanol Project Quality Assurance Plan (QAP).

3. References

3.1 TAPPI Test Method T211, "Ash in Wood and Pulp." *In Tappi Test Methods.* Atlanta, GA: Technical Association of the Pulp and Paper Industry.

3.2 Moore, W., and D. Johnson. 1967. *Procedures for the Chemical Analysis of Wood and Wood Products*. Madison, WI: U.S. Forest Products Laboratory, U.S. Department of Agriculture.

4. Terminology

4.1 Ash - The inorganic residue left after ignition at 575^{o}C.

5. Significance and Use

5.1 The ash content is an approximate measure of the mineral content and other inorganic matter in biomass.

5.2 The ash content is used in conjunction with other assays to determine the total composition of biomass samples.

6. Apparatus

6.1 *Aluminum weighing pans or crucibles, 50 mL* - If crucibles are used, platinum crucibles are preferred, but silica or porcelain crucibles may be used.

6.2 *Muffle furnace* - An electric furnace is recommended for igniting the wood sample. A furnace fitted with an indicating pyrometer, so that the desired temperature can be maintained, is preferable.

6.3 *Analytical balance,* sensitive to 0.1 mg.

6.4 *Desiccator*.

6.5 *Drying oven,* with temperature control of $105 \pm 2^{o}C$.

7 ES&H Considerations

7.1 Follow all applicable NREL Laboratory Specific Hygiene Plan guidelines.

8. Test Specimen

8.1 Test specimens suitable for analysis by this procedure are as follows:
- biomass feedstocks,
- pretreated biomass,
- the solids fraction of fermentation residues.

8.2 Samples must be dried at $105^{o}C$ according to the Laboratory Analytical Procedure #001, Determination of Total Solids and Moisture in Biomass, prior to ash analysis. Air-dried material can be used in place of $105^{o}C$ dried material, but the weight of the material must be corrected for its moisture content prior to calculating the ash.

8.3 The test specimen shall consist of approximately 0.5 to 1.0 g of sample obtained in such a manner to ensure that it is representative of the entire lot of material being tested. For $105^{o}C$ dried samples containing large particles or chunks, it is recommended that the sample be ground or milled to reduce the size of the large pieces to less then 1 mm in diameter. The sample is then redried at $105^{o}C$ prior to testing.

9. Procedure

9.1 Mark a pan or crucible with a unique identification using a porcelain marker, place it in the muffle furnace, and bring to constant weight by igniting at $575 \pm 25^{o}C$. Remove the pan or crucible from the furnace, cool to room temperature in a desiccator, and weigh to the nearest 0.1 mg. Record this weight as the tare weight. Keep the pan or crucible in a desiccator until used.

> Note: For an aluminum pan, two hours of heating at $575 \pm 25^{o}C$ will be sufficient to bring the pan to constant weight. With a crucible, however, the following procedure is used: Place the crucible in the furnace at $575 \pm 25^{o}C$ for three hours. Remove the crucible and place in a desiccator. Allow the crucible to cool to room temperature and then weigh the crucible to the nearest 0.1 mg. Record this weight. After weighing, return the crucible to the furnace for one hour at $575 \pm 25^{o}C$, cool again in the desiccator, and reweigh. Repeat this step until the weight of the crucible varies by less than 0.3 mg from the previous weighing. Record this final weight as the crucible tare weight.

9.2 Weigh approximately 0.5 to 1.0 g, to the nearest 0.1 mg, of a test specimen into the tared pan or crucible. If the sample being analyzed is a $105^{o}C$ dried test specimen, the sample should be stored in a desiccator until use. Record the weight (container plus sample minus tare weight of container) as the initial weight of the test specimen, W_2.

> Note: For air dried samples, we recommend that samples for moisture determination should be weighed out at the same time as the samples for the ash determination. If this is done at a later time it can introduce an error in the calculation because ground biomass can rapidly gain or lose moisture when exposed to the atmosphere.

9.3 Place the container and contents in the muffle furnace and ignite at $575 \pm 25^{o}C$ for a minimum of three hours, or until all the carbon is eliminated. Heat slowly at the start to avoid flaming. If the sample tends to flare up, the container should be partially covered during this step. Avoid heating above the maximum stated temperature. Protect the test container from strong drafts at all times to avoid mechanical loss of test specimen.

> Note: For test specimens containing high amounts of ash (greater than 5% by weight), it will be necessary to increase the time in the furnace to overnight to ensure complete elimination of the carbon. This ignition time period should not exceed 24 hours.

9.4 Remove the pan or crucible with its contents to a desiccator, cool to room temperature, weigh to the nearest 0.1 mg, and record this weight. Repeat the heating for one hour periods until the weight after cooling is constant to within0.3 mg. Record the final weight of the ash, W_1, as the container plus ash weight minus container tare weight.

10. Calculations

10.1 For 105°C dried materials, calculate the percentage of ash, based on the initial weight of the test specimen, as follows.

$$\text{Ash, \%} = (W_1/W_2) \times 100$$

where: W_1 = weight of ash, and
W_2 = initial weight of 105°C dried sample.

10.2 For air dried samples, the following calculation may be used to report the results on a 105°C dried weight basis:

$$\text{Ash, \%} = (W_1 / (W_2 \times T/100)) \times 100$$

where:

W_1 = weight of ash,
W_2 = initial weight of sample, and
T = percent total solids of sample, on a 105°C oven-dried weight basis, as determined by LAP #001.

11. Report

11.1 Report the result to two decimal places, as a percentage of the sample's 105°C dried weight, and cite the basis used in the calculation.

12. Precision and Bias

12.1 Data obtained by replicate testing of a hybrid poplar in one laboratory gave a standard deviation in ash content of 0.05% and a CV% of 3.88%. Replicate testing of a National Institute of Standards and Technology (NIST) #8494 wheat straw gave a standard deviation of 0.20% and a CV% of 1.95%.

12.2 Data obtained by replicate testing of a hybrid poplar sample in six different laboratories gave a standard deviation in ash content of 0.11% and a CV% of 9.24%

13. Quality Control

13.1 *Reported significant figures:* The ash results will be reported as a percentage with two decimal places.

13.2 *Replicates:* All samples and all method verification standards are to be analyzed in duplicate.

13.3 *Blank:* An empty aluminum dish or crucible is to be run through the analysis. The dish is to be weighed empty, ashed, and reweighed. The difference in weight must be less than the equivalent of a 0.5% error.

13.4 *Relative percent difference criteria:* The %RPD must be less than 15.5% if the average ash content is less than 2%. The %RPD must be less than 5.0% if the average ash content is greater than 2%. If the %RPD is too large, the sample will be rerun.

13.5 *Method verification standard:* A method verification standard must be run in duplicate with every batch. The results of method verification analyses must be control charted.

13.6 *Calibration verification standard:* Not applicable.

13.7 *Sample size:* Approximately two grams are required for duplicate analyses. If there is insufficient sample, the result will be flagged and the lack of precision will be noted.

13.8 *Sample storage:* The oven dried sample will be stored in a desiccator.

13.9 *Standard storage:* Not applicable.

13.10 *Standard preparation:* Not applicable.

13.11 *Definition of a batch*: Any number of samples which are analyzed together and recorded together. Samples within a batch must be of the same matrix. The maximum size of a batch would be limited by the equipment constraints. A batch cannot be larger than what is practical with the equipment.

13.12 *Control charts:* The results of analysis of method verification standards are recorded. Each result from the duplicate analysis is recorded along with the average, %RPD, and a laboratory book/page reference.

13.13 *Other:* All aluminum pans or crucibles shall be preashed. Because the samples and containers quickly pick up moisture from the air, all the weights shall be taken immediately after the items are removed from the desiccator.